

TECHNICAL SYSTEMS AUDIT CHECKLIST FOR SAMPLES COLLECTED DURING DISCHARGE

Purpose/Scope of Audit: GSI Research, Development, Testing, and Evaluation (RDTE) Facility Technical Systems Audit

Brief Description of Audit: Audit of sample labeling, collection, transport, and analysis at the GSI RDTE Facility during performance evaluation of the Siemens SiCURE Ballast Water Management System.

Auditee: GSI scientists

Audit Location: RDTE Facility (Superior, WI)

Auditors: Kelsey R. Prihoda, GSI Assistant Quality Assurance Manager

Audit Dates: Wednesday, September 16, 2009

SAMPLE BOTTLE LABELING, SAMPLE COLLECTION, AND SAMPLE TRANSPORT TO UWS

SAMPLE TEST ID: 09-SI-4D *Sample Bottles Labeled by KRP 9-15-09*

Relevant GSI SOPs:

- GSI/SOP/G/RA/SC/3 – Procedure for Labeling Samples Collected at the GSI Land-Based RDTE Facility (DRAFT)
- GSI/SOP/LB/G/O/5 – Procedure for Injecting Organisms and Solids into the GSI Land-Based RDTE Facility
- GSI/SOP/LB/RA/SC/3 – Procedure for Algae/Small Protozoa Sample Collection
- GSI/SOP/LB/RA/SC/4 – Procedure for Microbial Sample Collection
- GSI/SOP/LB/RA/SC/6 – Procedure for Zooplankton Sample Collection
- GSI/SOP/LB/RA/SC/3 – Procedure for Collecting Physical/Chemical Data and Samples at the GSI Land-Based RDTE Facility (DRAFT)

➤ **Time Discharge Treatment Tank Started:** *11:49 am* **Time Discharge Treatment Tank Completed:** *12:43 pm*
 ➤ **Time Discharge Control Tank Started:** *~8:56 am* **Time Discharge Control Tank Completed:** *9:52 am*

Sample Collection Type (Code)	Sample Port/Point	Tub Number	Sample Type (Collected By)	Labeled Correctly & In Crate?		Collected Following SOPs?		Transported Back to UWS?	
				Y	N	Y	N	Y	N
Control Tub (C)	SP9-C	1	• Phytoplankton	✓		✓	<i>9:52</i>		
			• Zooplankton	✓		✓	<i>9:53</i>		
			• Microbe Rep. 1	✓		✓	<i>9:53</i>	✓	
			• Microbe Rep. 2	✓		✓	<i>9:53</i>	✓	
			• Microbe Rep. 3	✓		✓	<i>9:53</i>	✓	
			• TRC and TRO	✓		✓	<i>9:54</i>		
			• Disinfection Byproducts (2 L)	✓		✓	<i>9:54</i>	✓	

Tom marker to trans-port.

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				Y	N	Y	N	Y	N
Pre-Treatment In-Line (PT)	SP10-C		TSS Rep. 1 Rep. 1 ~ 10 min. TSS Rep. 2 Rep. 2 ~ 30 min. TSS Rep. 3 Rep. 3 ~ 50 min.						
① See comments Treatment Tub #4 (T)	SP9-B	5	Phytoplankton - Rep. 1	✓		TUB 5 12:43			
			Zooplankton	✓		TUB 6			
			Microbe - Rep. 1	✓		TUB 5 12:44		✓	
			TRC and TRO - Rep. 1	✓		TUB 5 12:45			
			Phytoplankton - Rep. 2	✓		12:43			
Treatment Tub #5 (T)	SP9-B	5	Zooplankton	✓		Began at 12:57			
			Microbe - Rep. 2	✓		12:44		✓	
			TRC and TRO - Rep. 2	✓		12:45			
			Phytoplankton	✓		✓			
			Microbe	✓		✓		✓	
Treatment Tube #6 (T)	SP9-A	6	TRC and TRO	✓		12:48			
			TRC and TRO Duplicate	✓		12:48			
			Whole Effluent (~38 L)	✓		12:49		✓	
			Disinfection Byproducts (2 L)	✓		12:48			
Treatment In-Line (T)	LINE		TSS Rep. 1 ~ 10 min. TSS Rep. 2 ~ 30 min. TSS Rep. 3 ~ 50 min. TSS Rep. 3 ~ 50 min. Duplicate						
Treatment Tank 2 and 4	Mid-Depth		TRC Monitoring - Day 5 and 4	✓		✓			

Potable Water Tank - post-filtration from rinse hose

Microbe

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Initial/Date

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* There will be no TSS samples collected from the control or treatment lines upon discharge. We have proven during 09 SI 1-3 that the discharged water is below the TSS level set by GSI RSTC permits.

Unlabeled P.O.U. to heavy port

* Not part of test plan

* Not part of test plan

SAMPLE ANALYSIS

SAMPLE TEST ID: 09-SI-4D

QUALITY SYSTEM DOCUMENTATION

AUDIT QUESTIONS	RESPONSE			COMMENTS
	Y	N	NA	
* See 09 SI 4 Fill TQA checklist for responses to questions #1-10. All in the same for discharge.				
1. Is there an approved Quality Assurance Project Plan for the overall project and has it been reviewed by all appropriate personnel?				
2. Is a copy of the current approved QA Project Plan maintained near laboratory work station areas?				
3. Is the implementation of the project in accordance with the QA Project Plan?				
4. Are there deviations from the QA Project Plan? Explain.				
5. Do any deviations from the QA Project Plan affect data quality?				
6. Are sample handling and storage procedures in accordance with the QA Project Plan?				
7. Are written and approved current standard operating procedures (SOPs) used in the project? If so, list them and note whether they are maintained near laboratory work station areas?				
8. Are data/observations appropriately recorded in laboratory notebooks/forms according to the QA Project Plan (i.e., entries in ink, dated, initialed, corrections done properly)? Are data contained in bound, well-labeled notebooks or three-ring binders?				
9. Do supervisory and/or QA personnel inspect laboratory notebooks/forms on a regular basis and initial notebook after review?				
10. Are paper records written in indelible ink?				
Additional Questions or Comments:				

CHEMISTRY

Relevant GSI SOPs:

- GSI/SOP/BS/RA/C/2 – Procedure for Determining Total Residual Oxidants (TRO) in Water
- GSI/SOP/BS/RA/C/3 – Procedures for Measuring Organic Carbon in Aqueous Samples

- GSI/SOP/BS/RA/C/6 – Procedure for Analyzing Total Residual Chlorine (TRC) Concentrations in Water
- GSI/SOP/BS/RA/C/8 – Procedure for Analyzing Total Suspended Solids (TSS)

AUDIT QUESTIONS	RESPONSE			COMMENTS
	Y	N	NA	
1. Describe the analytical instrumentation. List the brand and model number for each instrument.				
2. Are calibration and maintenance logs kept for the instrumentation (e.g., balances and other equipment)?				
3. Review the maintenance and operational records for the equipment. Based on your findings, do all instruments/equipment appear to be in good operating condition?				
4. Are the manufacturer's operating manuals readily available to the instrumentation operators?				
5. Describe the routine calibration procedure.				
6. Does the calibration documentation show that the calibration procedures are being followed?				
7. Do the calibration standards have the appropriate levels (i.e., bracket the samples to be measured)?				
8. What is the instrumentation calibration error according to the calibration documentation?				
9. Are duplicate samples collected and analyses conducted on at least 10% of the physical/chemical samples?				
10. Are reagent blank samples analyzed with each set of samples?				
11. Are a minimum of three and preferably more standards required for standard curves?				
12. When applicable, do routine procedures that require standard curves bracket concentrations?				
13. When applicable, have analytical method detection limits been established and clearly documented?				
Additional Questions or Comments: TRC = 0.064 mg/L in Treatment Tank 2.				

MICROBIOLOGY

Relevant GSI SOPs:

- GSI/SOP/BS/RA/MA/1 – Procedure for Quantifying Heterotrophic Plate Counts (HPCs) using IDEXX's SimPlate® for

- GSI/SOP/BS/RA/MA/3 - Procedure for the Detection and Enumeration of Enterococcus using Enterolert™
- GSI/SOP/BS/RA/MA/4 - Procedure for the Detection and Enumeration of Total Coliforms and E. coli using IDEXX's Colilert®

AUDIT QUESTIONS	RESPONSE			COMMENTS
	Y	N	NA	
1. Are duplicate sample analyses conducted on at least 10% of the microbiology samples?	✓			1 DUP each for EC, ENT, HPC, VC-DNA, + VC-RNA!
2. Are at least 10% of the samples counted by a second qualified individual (i.e., QA count)?	✓			QA count done for EC and ENT, not VC or HPC.
3. Are reagent blank samples analyzed with each set of samples?	✓			Blanks for VC, HPC, ENT, + ECO. + Controls for ECO and ENT.
4. When applicable, have analytical method detection limits been established and clearly documented?	✓			1 = ECO, ENT VC = ? 2 = HPC
Additional Questions or Comments: Control samples collected by 9:53 am. Treatment samples collected by 12:44 pm. Microbiology analysis done @ ~2:00 pm (sample prep. and incubation). Control samples all in incubator by 12:39 pm. Treatment samples in incubator by 1:55 pm. Time of treatment neutralization finished by not recorded for EC, TC, ENT, and HPC. *Initial counts for EC, TC, ENT, and HPC + treatment samples done 2.75-1.25 hours earlier than directed by SOPs, because of this two "QA" counts had to be done (one is actually final count and one is QA count). <i>WMP checked one density calculation per species.</i>				

Relevant GSI SOPs:

- GSI/SOP/LB/RA/SA/1 - Procedure for Algae/Small Protozoan Sample Analysis

AUDIT QUESTIONS	RESPONSE			COMMENTS
	Y	N	NA	
1. Were all data, observations, and comments appropriately recorded on the "Ballast Water Plankton Count Sheet"?	✓			
2. Was sample assessment conducted within ~1-1.5 hours after sample collection?	✓			Treatment tank began @ 12:55 pm Done @ 1:17 pm
3. Were at least 10% of the samples counted by a second analyst (i.e., QA count)?	✓			QA count done during fill.
Additional Questions or Comments: - 193 live/mL in control tank. - 0 live/mL in treatment tank.				

ZOOPLANKTON

Relevant GSI SOPs:

- GSI/SOP/BS/RA/C/2 – Procedure for Zooplankton Sample Analysis (DRAFT)

AUDIT QUESTIONS	RESPONSE			COMMENTS
	Y	N	NA	
1. Were all data, observations, and comments appropriately recorded on the "Zooplankton Identification Worksheet"?	✓	✓		
2. Was sample assessment conducted within ~2 hours after sample collection?	✓	✓		Tub 5 Rec'd at: 1:15 pm Tub 5 Done at: 2:15 pm
3. Were at least 10% of the samples counted by a second analyst (i.e., QA count)?	✓	✓		Tub 6 Rec'd at: 2:15 pm Tub 6 Done at: 3:02 pm QA count done on fill.
Additional Questions or Comments: $\sim 4 \times 10^5$ live organisms/m ³ in control tank sample. Control Discharge Tub 1 sample exceeded the 150-200 orgs. per subsample SOP directive for all rotifer and crustacean subsamples.				

WHOLE EFFLUENT TOXICITY (WET)/COLD WATER BIOASSAY (CWB) TESTING

Relevant GSI SOPs:

- GSI/SOP/BS/RA/RT/6 – Procedure for Assessing Chronic Residual Toxicity of a Ballast Treatment System to *Ceriodaphnia dubia*
- GSI/SOP/BS/RA/RT/7 – Procedure for Assessing Chronic Residual Toxicity of a Ballast Treatment System to the Fathead Minnow (*Pimephales promelas*)
- GSI/SOP/BS/RA/RT/8 – Procedure for Assessing Chronic Residual Toxicity of a Ballast Water Treatment System to the Green Alga (*Selenastrum capricornutum*) (DRAFT)

AUDIT QUESTIONS	RESPONSE			COMMENTS
	Y	N	NA	
1. Were all data, observations, and comments appropriately recorded on pre-printed data sheets and/or laboratory notebooks?	✓	✓		"Prepared By" column not filled out on stock sol-n prep. Algal counts done on notebook paper and transcribed into data sheet.
2. Were all relevant standard operating procedures followed (see above)?	✓	✓		* Sop deviations in comments.
3. Was an organism QA count done on at least 10% of the test chambers by a second, qualified analyst?	✓	✓		QA count done on 100% <i>C. dubia</i> test chambers and 100% of <i>P. promelas</i> test chambers.

#1 Comments (cont): Day 1-100% nothing recorded for "number surviving *C. dubia*".

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Additional Questions or Comments:

Day 0 Dissolved Oxygen values had to be adjusted by 1.8 mg/l because the meter was not properly maintained (air bubbles in probe).

* *C. dubia* acclimated for 6 hours only, not 24 hours. → SOP deviation
C. dubia added to 6.25% trt. at 9:34 am and 0% trt. reps. 9 and 10 added at 9:30 am. All others added starting at 2:30 pm, this must be a recording error. Need to clarify with MTEHP.

* SOP Deviation: Average Initial Cell Density = 102,679 → this is 10.3x higher than directed in GSI SOP and EPA WET methods.

Additional Questions and Comments on Technical Systems Audit:

- Drained control (C2) tank first in order to have a place to put recirculated water from treatment (T2) tank.

- Prior to drain/recirculation of T2 potable water from tanker truck was used to fill shared lines used by control and treatment drains. This water was purged out of SP9 pitot. The water flow rate is not fast enough to be classified a true "flush".

- The sample lines going to Tubs 4, 5, and 6 were replaced with clean, flexible PVC that can be removed and cleaned after each use.

- Zooplankton nets were verified to be clean 15 Sept. 09.

① - At ~15 sec. prior to treatment tubs filling, DMR noticed very dark water entering Tub 4. Due to possible contamination in Tub 4 no samples will be collected from this tub. Zooplankton will be collected from Tub 6, and all other samples were collected from Tub 5.